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AQUEOUS EXTRACTION OF THE FRUIT OF *Rosa canina*

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The optimum degree of grinding, temperature, and ratio between raw material and extractant for the aqueous extraction of the extractive substances, including the total organic acids, from the fruit of the dog rose have been determined by a monofactorial experiment under static conditions.

The fruit of the dog rose is a well-known medicinal raw material which includes vitamins, organic acids, flavonoids, carotenoids, macro- and microelements, and various lipids [1]. Aqueous extracts of the fruit (tinctures, decoctions, syrups) are used in folk and traditional medicines for the treatment of urolithic diseases and affections of the liver and bladder, and also as vitamin preparations for preventing capillary fragility; in addition, they exhibit a weak hemostatic action [2].

The level of biologically active extractive substances (ESs), including total organic acids (TOAs) in aqueous extracts of dog rose fruit, varies within wide limits and depends on the quality of the raw material and the conditions of extraction.

In order to choose the optimum conditions for extracting the water-soluble ESs, including the TOAs, we have investigated the influence of the degree of grinding of the raw material, the extraction temperature and the ratio between extractant and raw material on the yield of ESs and have studied the dynamics of this process.

We extracted fruit with a degree of grinding of from 2 to 8 mm under static conditions (Table 1). On grinding to a particle size of less than 2 mm, not only the flesh but also the seeds are broken down, which is undesirable. Furthermore, the degree of agglomeration of the particles increases, and this has a negative effect on the yields of ESs and TOAs.

It can be seen from Table 1 that with a decrease in particle size the yield of final product, in which we qualitatively determined the presence of carbohydrates, pectin substances, vitamin C, flavonoids and malic and citric acids, increased by 18.0%. Subsequent experiments were therefore carried out with raw material ground to 2-4 mm.

To choose the optimum temperature, we used the interval of 50-90°C (Table 2). It can be seen from Table 2 that raising the temperature from 70 to 90°C was accompanied by an insignificant increase in the yield of ESs and TOAs, and we therefore took 70-80°C as the optimum.

In a study of the relationship between the amounts of extractant and raw material, the weight fraction of extractant was chosen in such a way as to ensure the most complete extraction of the desired product without unjustifiable consumption of extractant and increases in the duration of the process and in the consumption of energy for drying the extract. Extraction at 70-80°C and with a degree of grinding of the raw material of 2-4 mm was carried out at liquor ratios of 1:4, 1:7, 1:9, 1:12, and 1:14, allowing for the water absorption factor (Table 3).

It can be seen from Table 3 that in a single extraction the highest yields of ESs and TOAs was observed at a liquor ratio of 1:14. It is known that it is possible to achieve more complete exhaustion of the raw material with a smaller amount of extractant by multiple extraction [3]. In view of this, we carried out a threefold extraction of the fruit at ratios of 1:4, 1:3, and 1:2 (total ratio 1:9) and obtained yields of ES of 95.5% and of TOAs of 96.9%.

The wide use of the extraction of plant raw material in industry requires a careful study of the dynamics of the process. There is information in the literature on the dynamics of the process in various methods of extraction [4-6]. However, the dynamics of extraction depends substantially on the type of raw material. We have determined the time required for the most complete exhaustion of the raw material with three phase contacts and various ratios of raw material and water and have established the moment of phase equilibrium by a known procedure [7] (Table 4).

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TABLE 1. Influence of the Degree of Grinding of Dog Rose Fruit on the Yield of ESs and TOAs

Particle size, mm	Yield, % on amount in the raw material	
	ESs	TOAs
Unground	52.1	55.4
6–8	77.7	80.6
2–4	96.0	98.8

TABLE 2. Influence of the Temperature of Extraction of Dog Rose Fruit on the Yields of ESs and TOAs

Extraction temperature, °C	Yield, % of amount in the raw material	
	ESs	TOAs
50	64.2	66.0
60	87.2	89.4
70	95.0	97.0
90	95.8	97.5

TABLE 3. Influence of the Liquor Ratio on the Yields of ESs and TOAs

Liquor ratio	Yield, % of amount in the raw material	
	ESs	TOAs
1:4	65.0	67.7
1:7	87.3	89.4
1:9	91.2	93.0
1:12	94.6	96.1
1:14	96.5	98.0

It can be seen from Table 4 that to reach the equilibrium concentration of ESs at the first phase contact required 2.5 h, while phase equilibrium at the second contact was achieved after 2 h, and at the third after 1.5 h. For the TOAs, phase equilibrium set in after 2, 1.5, and 1.0 h, respectively. The extraction curves represented typical isotherms tending towards equilibrium (Fig. 1).

As was to be expected, with a decrease in the level of ESs and TOAs in the meal the relative rate of extraction fell, as the curves in Fig. 2 show.

The results that we obtained show that a threefold extraction by water of dog rose fruit with a degree of grinding of 2-4 mm at a total liquor ratio of 1:9 and a temperature of 70-80°C permits the practically complete extraction of the water-soluble substances.

EXPERIMENTAL

ESs were determined gravimetrically and refractometrically [8], and the sum of the TOAs titrimetrically [9]. We used dog rose fruit gathered in September, 1995 in the Samarkand oblast. The quality of the raw material satisfied pharmacopoeial requirements [8]. The fruit was ground in a laboratory mill with apertures having a diameter of 2 to 8 mm.

Extraction was conducted in 20-liter extractors with steam heating under static conditions at 50-90°C and liquor ratios of 1:7-1:14.

The dynamics of the extraction process was studied by establishing the phase equilibrium at first phase contact, for which purpose, 1 kg of the ground raw material was charged into each of six extractors with external heating. The process was conducted at 70-80°C. In the first extractor, the time of extraction was 0.5 h; in the second, 1 h; in the third, 1.5 h; in the fourth, 2 h; in the fifth, 2.5 h; and in the sixth, 3 h. After the lapse of these times the extracts were poured off, cooled, and analyzed.

For the second phase contact, 1 kg of ground raw material was extracted in each of five extractors for 2.5 h. The extracts were poured off, and fresh portions of extractant were added. The solution was poured off from the first extractor after 0.5 h, from the second after 1 h, from the third after 1.5 h, from the fourth after 2 h, and from the fifth after 2.5 h, and was subjected to analysis.

TABLE 4. Dynamics of the Extraction of ESs and TOAs

Time of extraction, h	Liquor ratio	Yield, % of amount in the raw material	
		ESs	TOAs
First contact of the phases after			
0.5		13.0	18.4
1.0		15.8	20.1
1.5		18.2	21.3
2.0	1:4	19.4	22.7
2.5		20.4	22.7
3.0		20.4	–
Second contact of the phases after			
0.5		6.1	5.0
1.0		7.6	6.6
1.5	1:3	8.6	7.6
2.0		9.3	7.6
2.5		9.3	–
Third contact of the phases after			
0.5		1.9	2.7
1.0		2.6	3.0
1.5	1:2	3.2	3.0
2.0		3.2	–

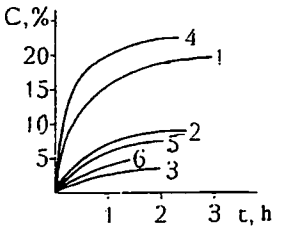

Fig. 1. Changes in the concentrations of water-soluble substances with time (1-3 extractions): for ESs — *1-3*; for TOAs– *4-6*.

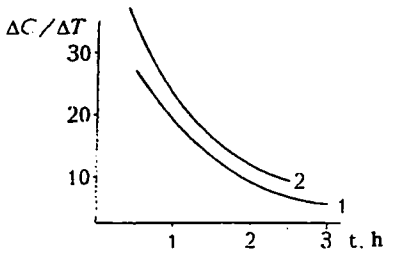

Fig. 2. Change in the rate of extraction of ESs (*1*) and TOAs (*2*) with time.

To determine the time of extraction at the third phase contact, 1 kg of ground raw material was extracted for 2.5 h in each of four extractors. The extracts were poured off, fresh solvent was added and the raw material was reextracted for 2 h. Then the time required to reach phase equilibrium at the third phase contact was determined.

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